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ROYAL CANADIAN MINT PILOT PLANTS FOR SOLVENT EXTRACTION OF COPPER

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by

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ABSTRACT

The Royal Mint wishes to recover platinum and palladium from their spent gold electrolyte. In order to carry out the precipitation of these metals, copper in the solution must be removed. A solvent extraction process has been developed to treat the electrolyte to extract copper. The process has been successfully tested on a pilot plant scale using real solutions.

AMÉNAGEMENT D'USINES-PILOTES PAR LA MONNAIE ROYALE CANADIENNE POUR L'EXTRACTION DU CUIVRE PAR SOLVANT

par

R. Molnar

RÉSUMÉ

La Monnaie royale canadienne projette de récupérer la platine et le palladium de l'électrolyte d'or usé. Pour que la précipitation de ces métaux puisse se produire, le cuivre doit être extrait de la solution. Un procédé d'extraction par solvant a été mis au point pour le traitement de l'électrolyte en vue d'en extraire le cuivre. L'essai du procédé à l'échelle-pilote avec des solutions réelles a été fructueux.

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INTRODUCTION

The Royal Mint in Ottawa operates a gold refinery. They produce, from their gold electro-refining circuit, a spent chloride electrolyte which contains principally, gold, platinum, palladium and copper. The gold is precipitated with SO_2 and the platinum and palladium are presently isolated by ion exchange. The ion exchange step is costly because the loaded resin must be burned and the ash sold for further recovery of the metal values. The Mint has been looking for ways to replace the ion exchange step to enable them to recover a more valuable product at lower cost.

The Mint solutions contain approximately 10 g/L copper. Precipitation of platinum and palladium by sodium formate is the technique being investigated by the Mint because it yields a high purity metal product. However, the copper which is present in greater amount than Pt and Pd interferes with the precipitation process and results in the loss of precious metal values.

The objective of this work is to study the possibility for the extraction of copper away from the Pt and Pd. The copper could be recovered in a concentrated solution and the Pt and Pd could be jointly precipitated from the solvent extraction raffinate with a minimum of impurities. An alternative approach, the selective extraction of platinum and palladium away from the copper is discussed separately.[1] Extraction of copper is a simpler process but the penalty is that the precious metals are not separated.

EXTRACTION OF COPPER

BACKGROUND

The technology of copper extraction from sulphate solution by solvent extraction has been well established.[2] More recently, work has been done on extraction of copper from chloride systems. Acorga P-5100 type extractant (Acorga Ltd. division of ICI) has been found to be effective in both sulphate[3] and chloride[4] systems. However, this reagent requires a pH at or above 2 to extract the copper. Stripping takes place at higher acidity. Acorga Ltd. has also been working on the development of an extractant for

more acidic conditions in a chloride environment. This reagent called DS-5443 is still in the experimental category.[5]

It is desired to extract the copper from the Mint liquor and then to precipitate metallic Pt and Pd with sodium formate. This precipitation has to be carried out around pH 6 so it must be preceded by neutralization. The as-received Mint solution contains about 3N HCl. Two treatment options were available. If possible, Cu could be extracted from the as-received liquor using DS-5443 and the pH then adjusted for precipitation. A schematic of this option is shown in Fig. 1. Alternatively, the pH could be adjusted to 2 prior to extraction, the copper extracted by Acorga P-5100, and the final pH adjustment made after extraction. This is illustrated in Fig. 2. Both of these flowsheet options were investigated.

EXPERIMENTAL

All bench scale experimentation was carried out with separatory funnel shake-outs at room temperature. The pilot plant was operated at CANMET using raffinate from the Pt/Pd circuit and at the Mint using real feed solution. The Bell Engineering type mixer-settler bank was used. All samples were analyzed at CANMET by atomic absorption spectrophotometry. Reagent grade chemicals were used except for the commercial grade extractants and diluents.

RESULTS AND DISCUSSION

Extractant Selection

The two extraction options were tested with series' of shake-outs over a range of phase ratios using two minute contacts at room temperature. The extractants tested were 5 v/o Acorga DS-5443 in Varsol 140 (Exxon) and 5 v/o Acorga P-5100 in the diluent. For the DS-5443, the aqueous feed was as-received Mint liquor. With the P-5100, the Mint liquor was adjusted to pH = 2 with NaOH before extraction.

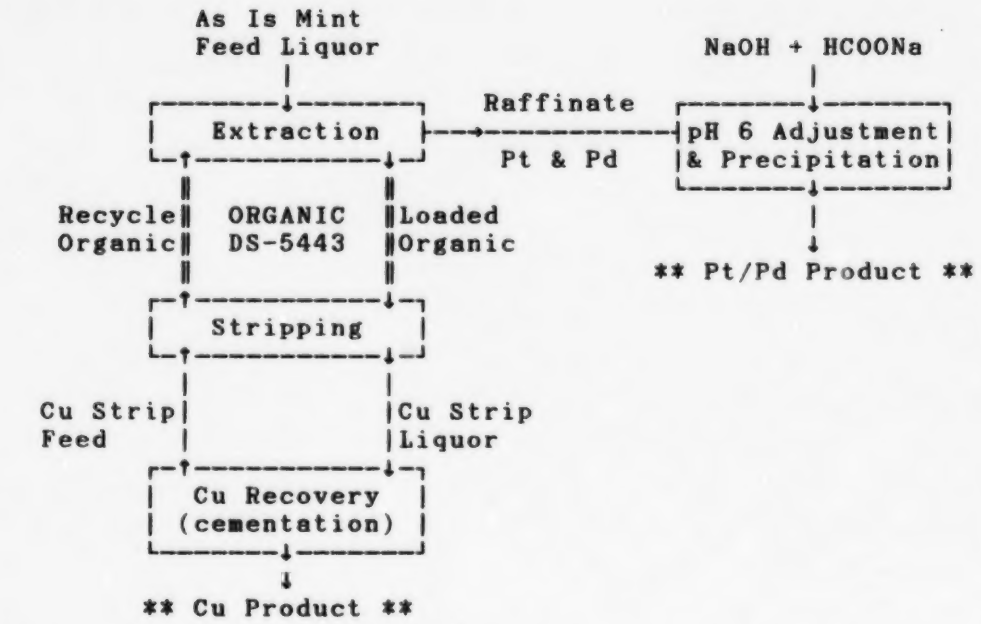


Figure 1 Copper Recovery by Direct Extraction

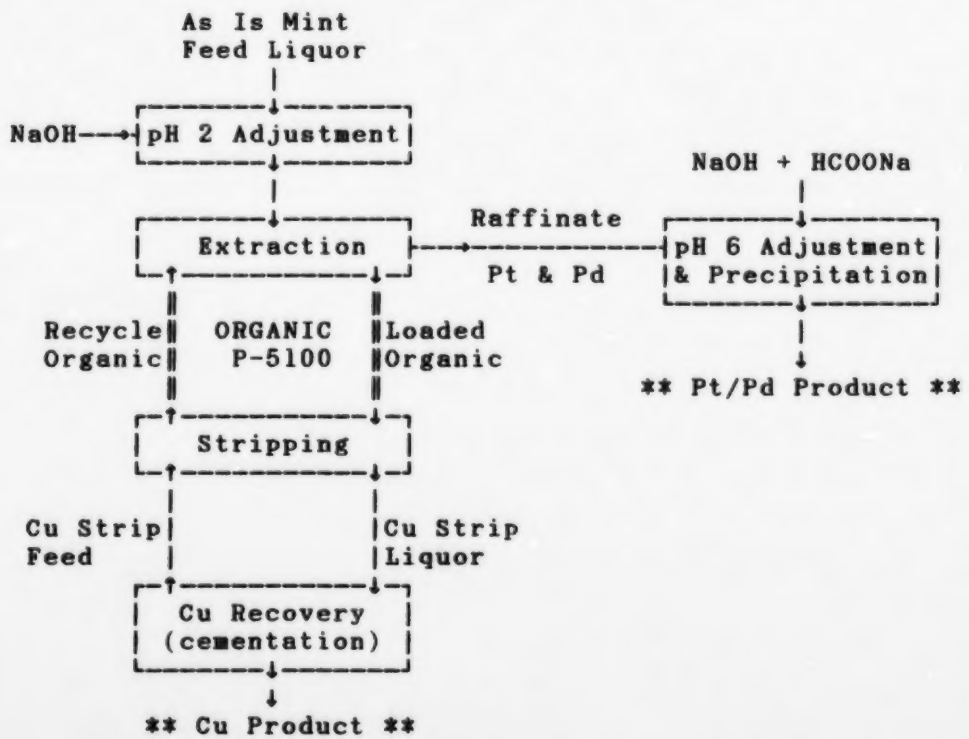


Figure 2 Copper Recovery by Extraction After pH Adjustment

There was effectively no copper extraction with the DS-5443. There was however, some evidence of Pd extraction. The Acorga P-5100 showed extraction of copper very much as expected from the manufacturer's literature.[6] Therefore, all subsequent testing was with the Acorga P-5100. Other reagents were not evaluated at this time. The P-5100 showed good phase separation behaviour with only a small amount of interfacial precipitation at O/A = 20. Phase disengagement times decreased from 90 sec. to 30-40 sec. as the phase ratio was increased from A/O = 1/20 to 2/1. The extraction results are presented in the form of an equilibrium isotherm in Fig. 3.

In an effort to increase the copper loading, the effect of using 10 v/o P-5100 was also studied and the resulting equilibrium isotherm is included in Fig. 3. Physically, the system operated well with clear phase separations (some haziness in the aqueous above A/O = 3) and phase disengagement times of 60-70 seconds. An increased loading of copper was attained and the isotherm is typical of results obtained in the sulphate system. The higher loading capacity of the stronger organic phase enables the use of fewer stages and a physically smaller circuit to achieve the same raffinate. Therefore 10 v/o was selected as the extractant for pilot plant testing.

Scrubbing of Pt and Pd

The extraction isotherm shake-outs indicated that some Pt and Pd were also being picked up by the organic phase. The DS-5443 showed the strongest tendency with a loading of 0.63 g Pd/L on the organic at O/A = 1/1. The P-5100 reagent was much more selective as indicated by the values in Table 1. Furthermore, with the copper loading at 10% P-5100 close to double that at 5% while the impurity loading remained the same, it can be said that the selectivity of the 10% reagent was double that of the 5% with twice the separation.

It was nevertheless deemed useful to conduct some preliminary screening tests for scrubbing agents which could selectively remove the small amount of Pt and Pd on the organic to prevent its being lost with the Cu. Room temperature shake-outs of 2 min. duration at A/O = 1 were used. The following reagents were found to be totally ineffective: 3.5N NaCl, 20 g/L Cu as $CuCl_2$ adjusted to pH = 2.5 with HCl, 3.5N NaCl + 0.5 v/o Formic Acid (pH as is = 1.62), 20 g/L Cu as $CuCl_2$ + 5 g/L Ammonium Formate (pH as is = 3.2), tap water to pH = 4.5 with HCl, and tap water as is (pH = 6). The

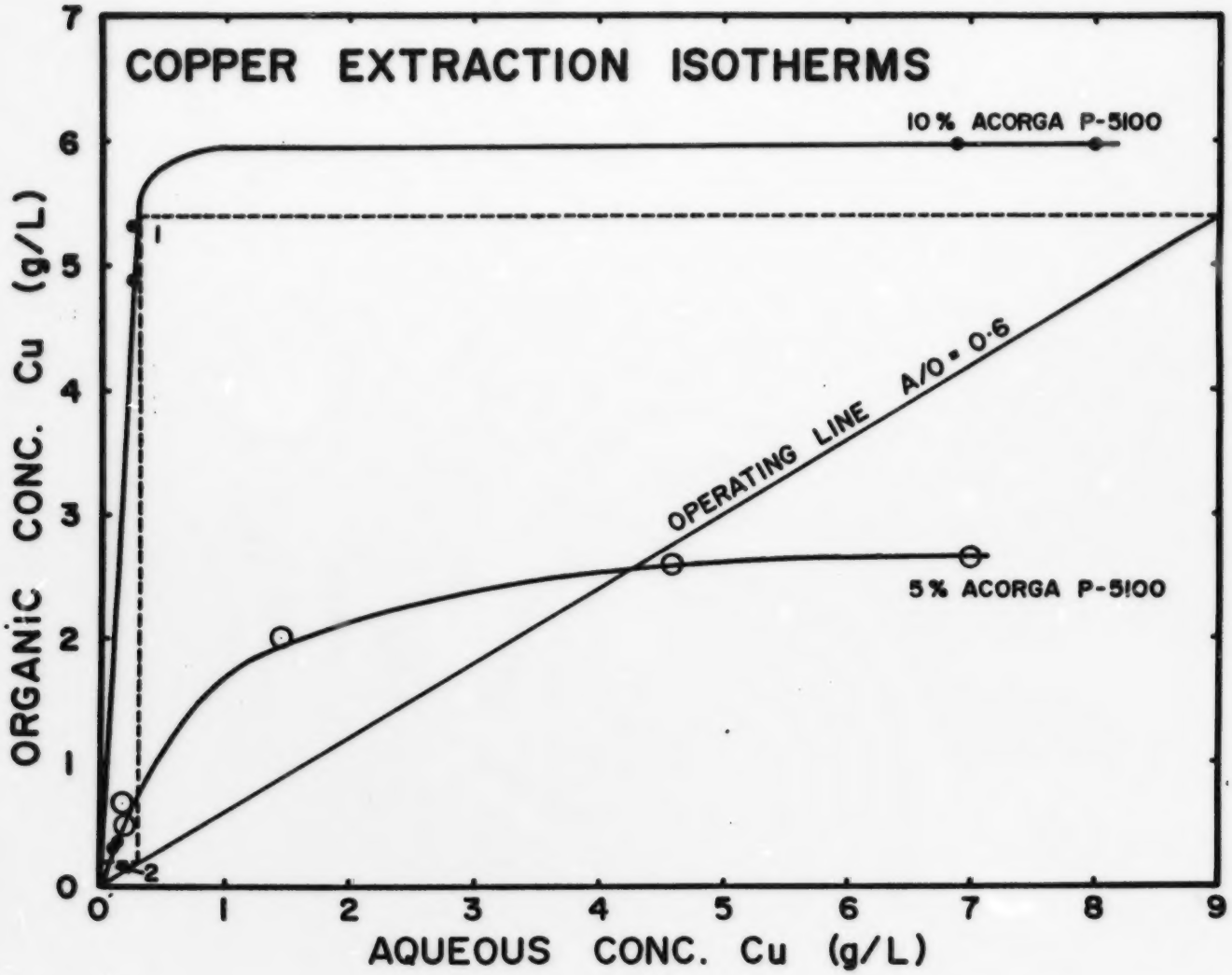


Figure 3. Copper Extraction Isotherms

Table 1. Impurity Concentrations in Organic Phase

	5 v/o DS-5443		Acorga P-5100			
			5 v/o		10 v/o	
O/A	Pd(ppm)	Pt(ppm)	Pd(ppm)	Pt(ppm)	Pd(ppm)	Pt(ppm)
1/30	93	10	17	<0.1	7	<60
1/10			21	0.5	14	<60
1/5					22	<60
1/3	240	9	14	0.1		
1/1	630	8	29	10	34	<60
3/1	840	10	28	6	28	<60
5/1					27	<60

final reagent tested, 8N HCl scrubbed over 70% of the Pd and Pt but it also stripped over 85% of the Cu and so was unacceptable. Furtner scrubbing tests were not carried out, but it would seem tnat a strong acid-strong copper solution (6N HCl, 25 g/L Cu) may well work.

Copper Stripping

Stripping of the loaded 5 v/o Acorga P-5100 was tried with different strengths of HCl. Two minute shake-outs at room temperature with O/A = 2/1 were used. The distribution of copper in the strip (aqueous/organic) changed from 10 to 234 to 59 as acid strength was increased from 50 to 100 to 150 g/L HCl. All Pt and Pd on the organic was completely stripped at all acid strengths. Therefore, 100 g/L was judged to be the optimum value and the equilibrium stripping isotherm shown in Fig. 4 was obtained for this strength of acid.

Pilot Plant Operation

Two pilot plant campaigns were operated. In the first at CANMET (RCMP-Cu-1), raffinate from the extraction of Pt and Pd by LIX 26 was used as the feed. In the second pilot plant at the Mint (RCMP-Cu-2), real feed solution was used. In both cases, the feed pH was adjusted with NaOH. There was considerable foam generation during the neutralization, together with precipitation, particularly in the second run. The precipitate was a beige/black mixture and was identified as consisting mainly of NaCl. It

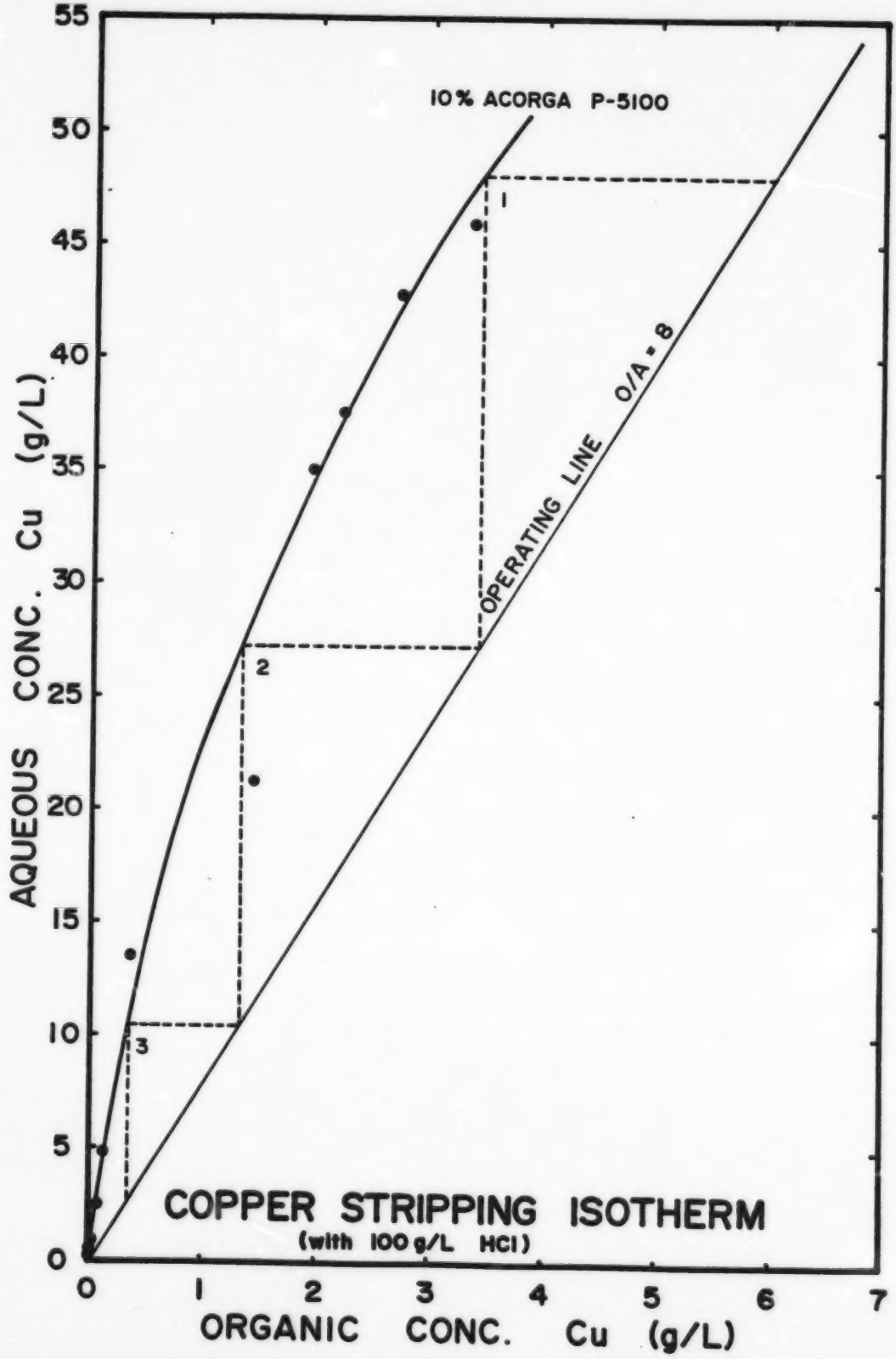


Figure 4. Copper Stripping Isotherm

was also thought that some of the Pt and Pd value was tied up on the precipitate. Most of the precipitation resulted from poor control during the neutralization with insufficient agitation. Addition of strong base causes local precipitation which then is very slow to redissolve. Any overshooting of the desired pH also adds precipitate which again is slow to redissolve even when the pH is corrected back down with acid.

Using the equilibrium extraction and stripping isotherms (Fig. 3 and 4), and the McCabe Thiele construction, it was estimated that 2 stages of extraction O/A = 2 and 3 stages of stripping at O/A = 8 would produce satisfactory results and these were the initially chosen conditions. The constructions are indicated on the figures. As the runs were carried out changes were made.

Table 2 lists the operating conditions for the two runs. Six samples sets were taken in each run and changes to conditions were made after sampling. The circuit was very small in RCMP-Cu-1 and it was estimated that the organic cycled through the 5 stages in about 1/2 hour. Therefore, samples were taken approximately two hours apart. For the RMCP-Cu-2 test, the organic cycle time was about one hour at most and samples were taken at roughly 2-1/2 hour intervals. As with the Pt/Pd circuit, aqueous continuous operating conditions were assumed throughout.

The pilot plants were the same in major details with alterations in flow rates and the number of stages as the table shows. In the second run an attempt was made to scrub any Pt and Pd which may have accumulated off the Cu stripped organic using strong acid. As well, for one sample period, a single water wash stage was added to the end of the circuit. This was done to prevent the loss of extraction efficiency which might take place if any of the scrub strong acid was entrained by the organic and returned to the extraction stage. The water wash was discontinued because it caused the organic to become cloudy showing poor phase separation in this step. Physically, the operation of the rest of the circuit in both runs was excellent. Both phases separated clearly throughout and there were no stable emulsions. No crud was observed. An in-line filter was used in the second run to prevent the precipitate in the feed from getting into the extraction circuit. A small amount of precipitate did get by the filter but this did not interfere with the operation.

Table 2. Copper Pilot Plants - Operating Conditions

RUN	SAMPLE SET / PARAMETER	1	2	3	4	5	6
RCMP-Cu-1	Sample time (hr)	2	4	5.5	7	9.25	12.5
	Extraction - Stages	2	2	2	2	2	2
	- O/A	1.6	2.0	2.4	2.5	2.5	3.0
	- Flows O/A (mL/min)	190/118	190/95	228/95	232/94	230/92	275/93
	- Res t mixers (min)	2.0	2.2	1.9	1.9	1.9	1.7
	- Res t settlers (min)	2.5	2.7	2.4	2.4	2.4	2.1
	Stripping - Stages	3	3	3	3	3	3
	- O/A	8.4	8.4	10.1	6.2	6.1	6.0
	- Flows O/A (mL-min)	190/22.5	190/22.5	228/22.5	232/37.5	230/37.5	275/46
	- Res t mixers (min)	2.9	2.9	2.5	2.3	2.3	1.9
	- Res t settlers (min)	3.7	3.7	3.1	2.9	2.9	2.4
RCMP-Cu-2	Sample time (hr)	3	5	7.5	10	12.5	15.25
	Extraction - Stages	3	3	3	3	3	3
	- O/A	2.5	2.5	2.2	2.2	2.2	2.3
	- Flows O/A (mL-min)	250/100	250/100	240/110	240/110	240/110	250/110
	- Res t mixers (min)	1.8	1.8	1.8	1.8	1.8	1.7
	- Res t settlers (min)	2.2	2.2	2.2	2.2	2.2	2.2
	Stripping - Stages	4	4	4	4	4	4
	- O/A	6.0	6.0	5.9	8.0	8.0	8.1
	- Flows O/A (mL/min)	250/42	250/42	240/40.5	240/30	240/30	250/31
	- Res t mixers (min)	2.1	2.1	2.2	2.3	2.3	2.2
	- Res t settlers (min)	2.7	2.7	2.8	2.9	2.9	2.8
	Scrub & Wash* - Stages				1*	2	2
	- O/A				8.0*	10.4	20.0
	- Flows O/A (mL-min)				240/30*	240/23	250/12.5
	- Res t mixers (min)				2.3*	2.4	2.4
	- Res t settlers (min)				2.9*	3.0	3.0

*Wash was carried out in conjunction with sample set 5

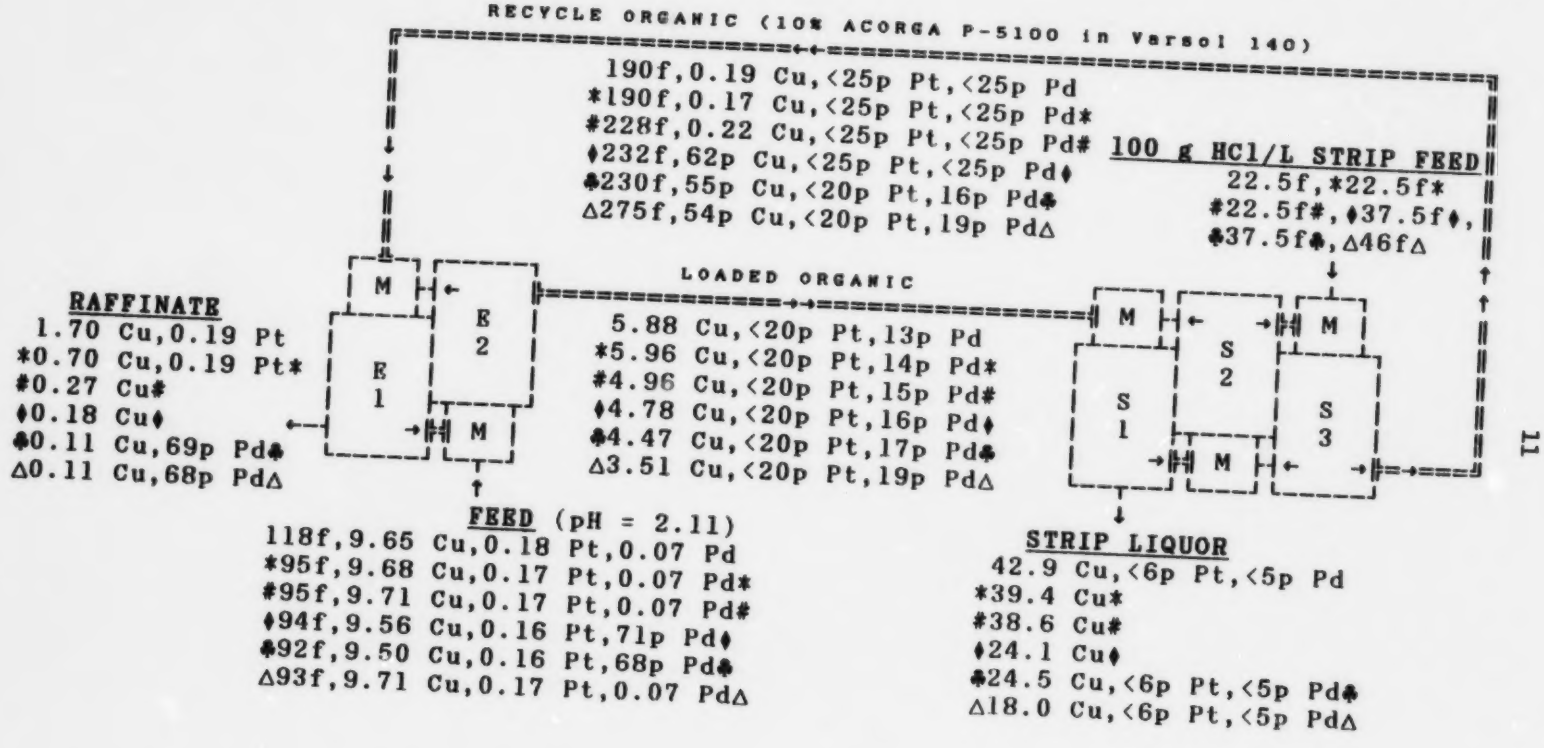
The chemical results of the two runs are indicated in Fig. 5 for RCMP-Cu-1 and Fig. 6 and 7 for RCMP-Cu-2. In the first run, the O/A ratio in extraction was increased from the initial value because the raffinate copper was not low enough. The O/A ratio in stripping was decreased after the third sample set to increase the amount of copper stripped off the loaded organic. This did have the desired result but it also diluted the strip liquor. The results of this first run did not give a picture of the role of Pt and Pd because the feed solution was depleted in those elements, being a raffinate from the previous tests. It was concluded that additional stages would be needed to decrease the raffinate copper and increase the concentration in the strip liquor.

The second run with real feed solution was carried out with three extraction and four stripping cells. This resulted in an order of magnitude decrease in the raffinate copper to the 10-20 ppm range. The stripping was also more effective as seen from the lower copper analyses in the recycle organic. It should be possible to keep the raffinate close to 10 ppm Cu with 3 stages of extraction at O/A $\geq$ 2.5 or this can be further lowered with the addition of another stage. With stripping at O/A $\approx$ 7 a strip liquor with 25 g Cu/L can be expected. Higher values are possible with the addition of more stages, and the equilibrium isotherm shows that values of 50 g/L should be attainable.

There was some evidence of a slight problem with Pt and Pd co-extraction. The organic Pt analyses were not sensitive enough to show up any trends but Pd was seen to be building up on the organic. There was also a consistent 10-20 ppm Pt and 20-40 ppm Pd in the strip liquor in the first four samples. When the strong acid scrub was added after the fourth sample, a sharp decrease was noted in the Pd level in the recycle organic showing that this was an effective measure. The effect on Pt could not be ascertained because of poor analytical sensitivity. The strong acid step did not seem to affect the extraction because the raffinate Cu was unchanged when the water wash step was discontinued after sample 5. The strip liquor composition did show a small decrease in impurities in the 5th sample with no effect on copper. The low copper value in the final strip liquor sample is not accurate as it does not check out with a mass balance on the aqueous circuit streams. Increasing the scrub O/A ratio decreased the effectiveness of the scrub as expected while producing a somewhat more concentrated scrub

Figure 5

Results of RCMP-Cu-1 Pilot Plant for Copper Extraction from Mint Liquor



SAMPLE SETS 1 (2 hr),*2* (4 hr),#3# (5.5 hr),♦4♦ (7hr),
♣5♣ (9¼ hr) and Δ6Δ (12.5 hr)

All Analyses in g/L or p = ppm = mg/L.
All Flowrates in mL/min designated by an "f".
=== = organic flows, --- = aqueous flows.

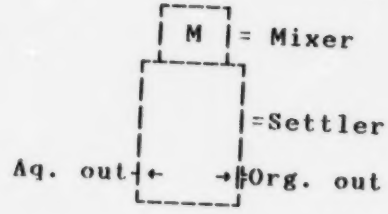
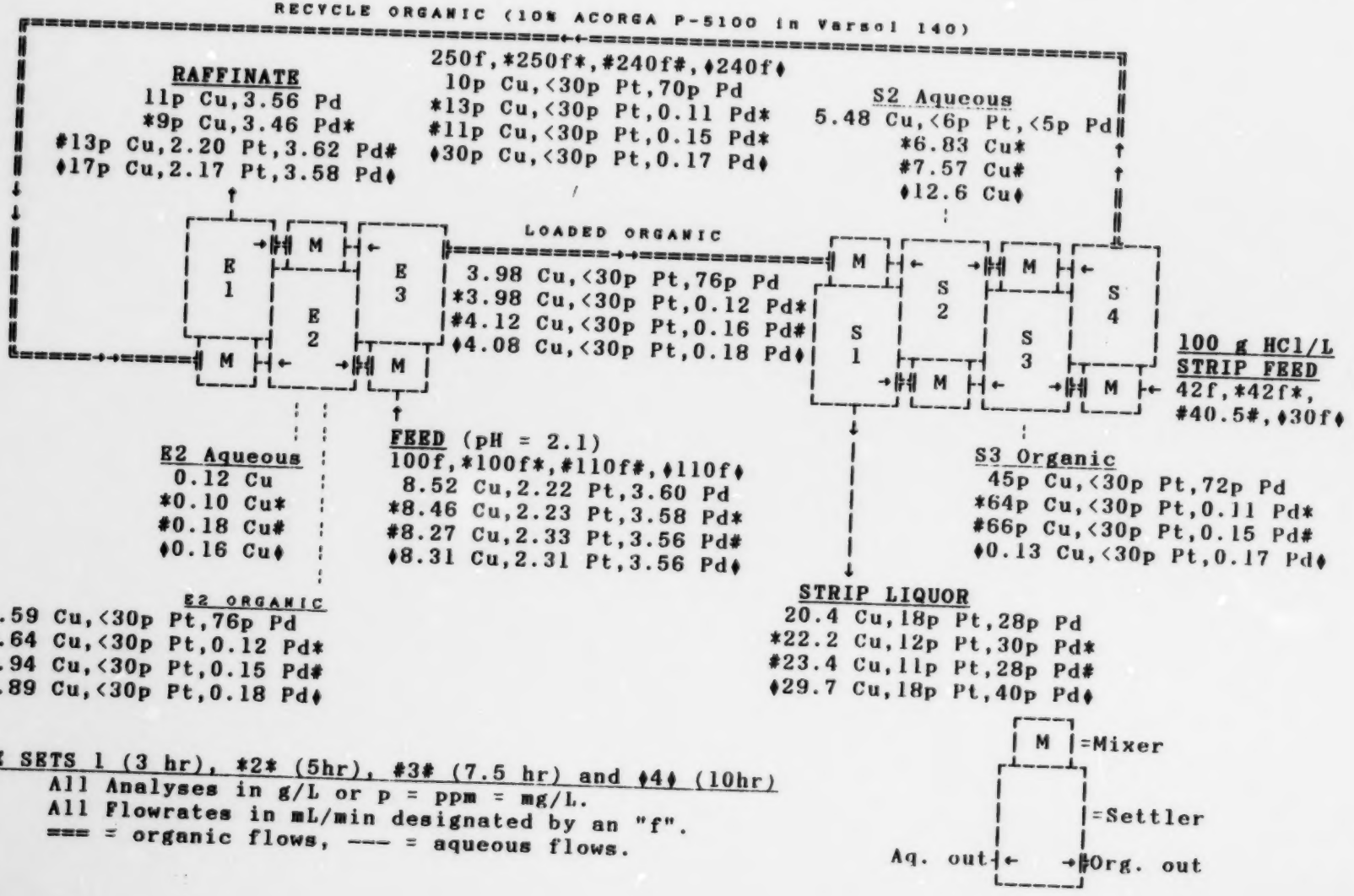


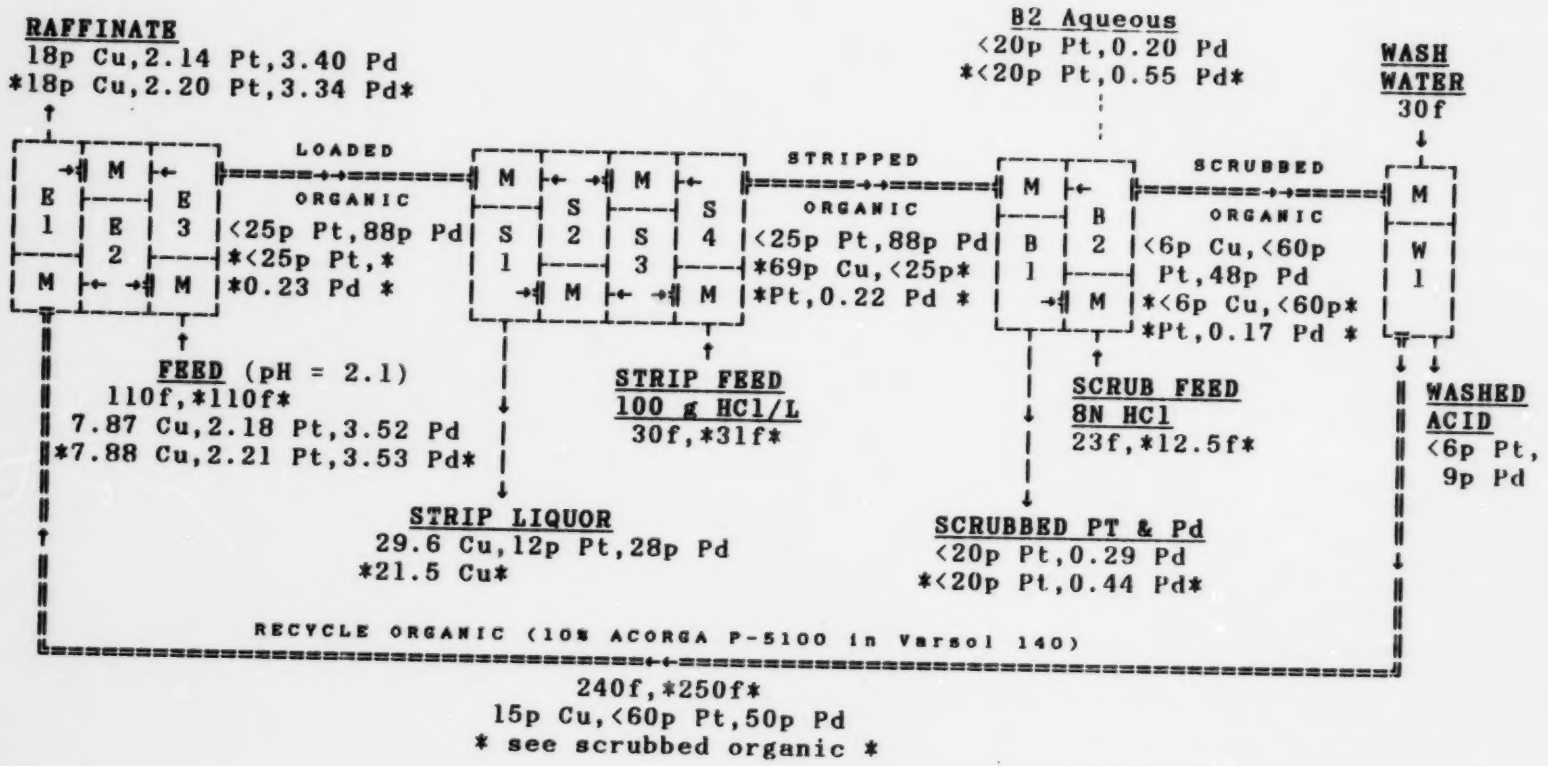
Figure 6 **Results of RCMP-Cu-2 Pilot Plant for Copper Extraction from Mint Liquor**



SAMPLE SETS 1 (3 hr), *2* (5hr), #3# (7.5 hr) and ♦4♦ (10hr)

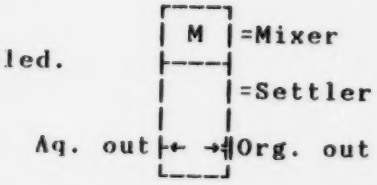
All Analyses in g/L or p = ppm = mg/L.
All Flowrates in mL/min designated by an "f".
=== = organic flows, --- = aqueous flows.

Figure 7 **Results of RCMP-Cu-2 Pilot Plant for Copper Extraction from Mint Liquor**



SAMPLE SETS 5 (12.5 h) and *6* (15¼ hr)

No water wash for sample set 6, scrubbed organic recycled.
All Analyses in g/L or p = ppm = mg/L.
All Flowrates in mL/min designated by an "f".
=== = organic flows, --- = aqueous flows.



liquor. Copper will also be scrubbed by the strong acid so if this step is to be used with the objective of returning the scrub liquor to the raffinate, a very effective copper strip is needed. It would seem that a more desirable solution to the co-extraction problem would be a scrub for Pt and Pd before copper stripping, perhaps with a strip liquor bleed which could be returned to extraction.

During the operation of the second pilot plant a sample of the raffinate was taken for a trial of the formate precipitation of Pt and Pd. When the pH of the solution was raised to 4 a red/brown material precipitated. This was subsequently identified as ferric hydroxide. Analysis of the precipitate at the Mint gave the following results: 12.25% Fe, 1.25% Pt, .85% Pd, .2% C with traces of Zn, Mn, Mg, and Ag based on the dry weight of precipitate. The results show that this precipitate could result in substantial losses of Pt and Pd if it has to be separated before the formate step. It is not known what level of Fe could be tolerated in the formate precipitate product before impurity penalties would be incurred.

COSTS FOR COPPER SOLVENT EXTRACTION

An estimate has been made of the capital and operating costs for the solvent extraction part of the circuit. It is assumed that 6000 L/a of feed is generated for treatment in two 3000 L batches. The feed is adjusted to pH = 2 with NaOH before solvent extraction. A Krebs type pilot plant unit is to be used for extraction at O/A = 2.5 and stripping at O/A = 8. A maximum of ten stages, that is two units like the one at CANMET, will be required, constructed from Fibreglass with titanium stirrers. These units can handle 1 L/min of feed at the operating O/A so they would require 3000 minutes to treat each feed batch, that is about 2-1/2 days on 3 shift operation or 7 single shift days. The 24 hour operation is preferred because the cells operate better with a minimum of interruptions. There is no advantage in going to a smaller unit. The cost of the equipment would not be significantly reduced because of the economies of scale. Solvent inventory is not a major cost and the units would occupy an area no larger than 1.5 m x 2.5 m; a smaller unit would not substantially reduce space requirements. Additional cost savings are possible by using mixer-settlers fabricated at the Mint from materials such as welded polypropylene. All prices and costs are based on current values.

The initial reagent cost is:
Considering 10 cells, 5 extraction at 70% organic and 5 stripping at 90% organic with 16 L total volume per cell -

Total organic = 5 x 16L x 0.7 + 5 x 16L x 0.9 = 130 L

Organic is 10% ACORGA P-5100 at $30/L
+ 90% Varsol 140 at $0.60/L = $3.54/L

Total organic cost = $460.

This is a conservative value because the cells can be operated with much less organic and more aqueous in the settlers where most of the volume/cell (13 L of 16 L) is.

The major capital cost items are:

1)	Krebs type mixer-settler units with stirrer motors, titanium stirrers, controls and all internal piping and connections.	- $14,000
2)	Aqueous feed pump - chloride resistant for 1 L/min.	- $ 800
3)	Aqueous strip feed pump - chloride resistant for 400 mL/min.	- $ 800
4)	Organic recycle pump - chloride and organic resistant for 2.5 L/min	- $ 1,000
5)	Holding tanks, 2 x 1200 L + 2 x 3200 L.	- $ 2,000
6)	Mixer for 3000 L feed tank for neutralization.	- $ 2,000
7)	Additional controls for automatic operation.	- $ 1,000
	TOTAL	= $21,600
	Contingency + installation + proper ventilation.	- $ 3,400
	TOTAL	= $25,000

Major operating costs per 3000 L cycle will be:

1)	NaOH to neutralize 3N HCl at \$2.90/kg - 3000 L x 3 moles NaOH/L x 0.04 kg/mole x 2.90/kg	= \$ 1,044
2)	Solvent losses at 1% of inventory (maximum)	= \$ 5
	TOTAL	= \$ 1,050

Not included are costs for Pt and Pd precipitation reagents, for disposal of the Cu strip liquor and the solution after precipitation of Pt and Pd, overhead costs, utility costs (only electric motors and water to wash up tanks) and labour costs (which should be about 1 man for 21 shifts).

Operating revenues will be:

3000 L x 2.2 g Pt/L = 6.6 kg Pt less ≈ 25 g Pt
+ 3000 L x 3.6 g Pd/L = 10.8 kg Pd less ≈ 50 g Pd

A revenue value taking \$400/troy oz Pt and \$130/troy oz Pd as typical recent prices would be about \$130,000.

Revenues will fluctuate with the metal prices and with the tenor of the feed solution. Losses will remain the same or lower with the refinement of operating conditions.

CONCLUSIONS AND RECOMMENDATIONS

From the results reported above it can be concluded that:

- the copper extraction circuit is economically viable, even at the small production rates required by the Mint.
- satisfactory extraction and stripping can be carried out with Acorga P-5100. There is sufficient flexibility in the system to enable conditions to be adjusted to produce raffinates as low in copper as desired.
- Acorga DS-5443 could not extract copper from the "as is" solution and it is necessary to raise the pH to around 2 before extraction with P-5100.

- the copper concentration in the strip liquor can be raised by adjustment of the operating conditions.
- a small problem remains with the co-extraction of Pt and Pd.
- an acid scrub stage after copper stripping can control the Pt and Pd levels on the recycled organic but this is not really effective in decreasing Pt and Pd losses in the copper strip liquor.
- the acid scrub step does not have any adverse effect on the copper extraction. A water wash stage to control the acid on the organic operates poorly and is not needed.

Further work may be required to decrease the Pt and Pd losses in the copper strip product. Optimization of the conditions should reduce co-extraction. Only two reagents were screened and other copper extractants may be more selective. Finally, scrubbing of the loaded organic may be possible.

Iron has to be traced through the circuit. It is possible that there is not enough iron to cause a problem if it is co-precipitated with the Pt and Pd. If the iron cannot be tolerated in the product there are a number of ways to prevent its coming out with the Pt and Pd. The simplest solution would be to prevent the Fe from precipitating out of the strip liquor when the pH is raised. Ferric ions precipitate above pH ≈ 2.5 while ferrous ions remain in solution until pH = 5.5.[7] If the iron in the raffinate can be reduced before the pH is raised and if the formate precipitation can take place below pH 5.5, the iron will remain in solution. The formate is a reducing agent so that if it is added before the raffinate pH is raised (at least enough added to reduce the solution) it may be possible to prevent precipitation of the iron.

The next simplest alternative would be to co-extract copper and iron with an organic extractant and it should be no problem to find a suitable reagent. Finally, it should also be possible to extract the iron from the raffinate leaving the Pt and Pd behind. This would produce a separate iron product. These different process alternatives are outlined in Fig. 8. Once it has been confirmed that an actual problem does exist with iron, the various solutions can be evaluated.

Finally, mention must be made of the most elementary option for the treatment of the spent electrolyte solution. This is the direct precipitation of the Pt and Pd with ammonia or ammonium hydroxide, the copper remaining in solution. Some preliminary work has been carried out at the Mint on this technique but the results have not been entirely satisfactory.[8] However, the extreme simplicity of this metnod demands that it be most thoroughly investigated before it can be rejected.

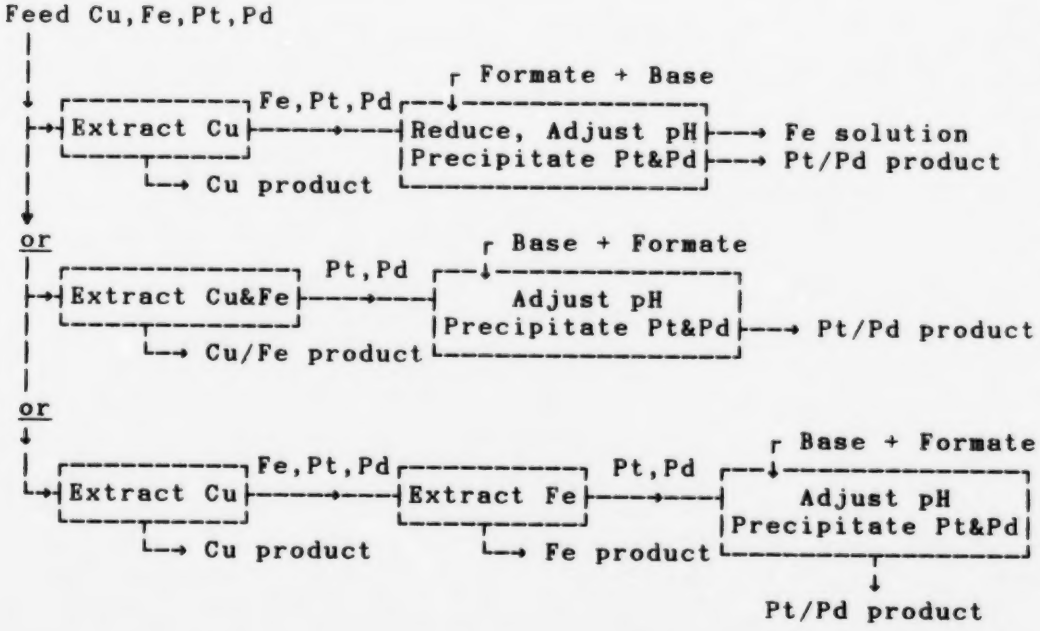


Fig. 8. Approaches to the Fe Problem

The expected revenues from Pt and Pd, even in a depressed market, exceed the capital and operating costs by such a large amount (over $120,000 vs less than $10,000 per cycle) for the different technology options that the decision on which to adopt should be based largely on which method will have the least environmental impact. Solvent extraction of copper has been

shown to be a technically and economically viable option even on a small scale, for the purification of the Pt, Pd solution prior to precipitation of these precious metals. It also provides a technological option which can keep liquid effluents to a minimum and reduce the pollution problems associated with the recovery of by-product precious metals.

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